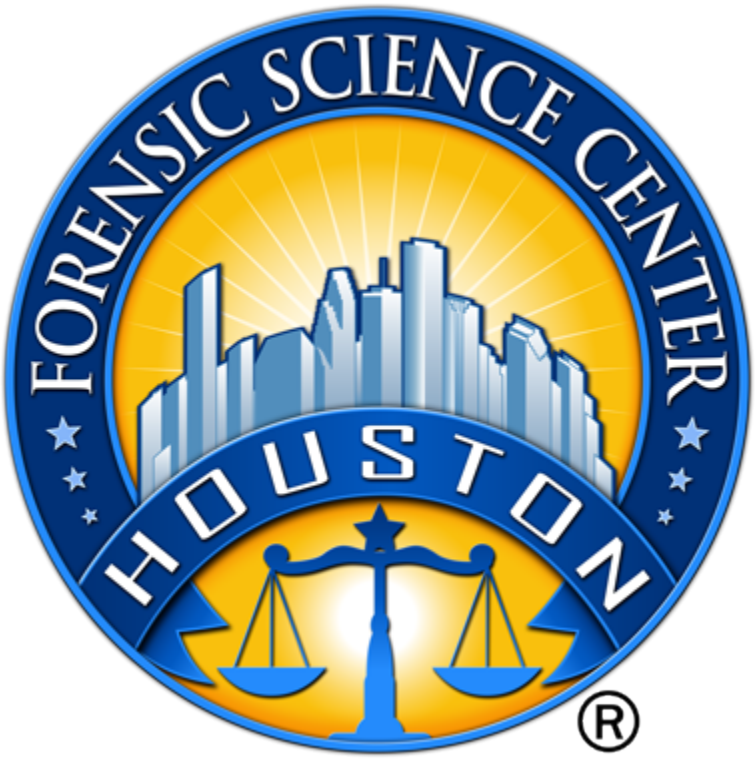

Seized Drugs
Operational Guidelines for the Thermo Nicolet iS50 FTIR_2

Comparative and Analytical Division

THERMO NICOLET iS50
FOURIER TRANSFORM INFRARED SPECTROMETER (FTIR_2)

Instrument

Nicolet iS50 Series FTIR spectrometer (Serial Number AUP1600099)
Smart iTR Diamond ATR Accessory

Software

OMNIC version 9.5.9 with ValPro version 2.7.1.45

Instrument Startup:

1. Turn on the computer and printer if not already on.

2. Ensure that the instrument is on by checking that the **System status** indicator light is blue. The **System scan** indicator light will flash blue with each scan of the interferometer. The instrument power should stay on at all times. If it has been turned off, turn it on by pressing the power switch. Let it stabilize for at least 15 minutes (one hour for best results) before collecting spectra.

3. On the computer desktop, open the software by double-clicking the **OMNIC** icon.

4. If the ATR accessory is installed (as for routine analysis), a window will appear to confirm the proper Experimental Method. Choose **"Smart iTR Diamond ATR (iTR Diamond.exp)"** from the drop-down menu and select **OK**.

5. The instrument will perform diagnostic tests, the results of which are indicated as "**System Status**" in the upper right corner of the window. If the indicator is a green check mark, the spectrometer has passed all of its diagnostic tests and is now ready to collect spectra. If the indicator is a red X, the spectrometer has failed a diagnostic test and requires corrective action before use. A message appears explaining the problem and allows access to information about correcting it. Record the results in the logbook.

6. Check the status of the internal desiccant by observing whether the indicator disc is blue or pink. If the indicator is blue, the desiccant is ok, if it is pink, then the desiccant should be replaced and documented. It is acceptable to use the instrument when the indicator is pink as the purpose of the desiccant is to prolong the use of internal components like the beamsplitter that are sensitive to the presence of water.

7. Align the spectrometer as follows:

   1.) While in **Experiment Setup**, Select the **Bench** tab and check that Gain = 1.

   2.) Be sure that there is no sample in the beam path and that the diamond is clean. Select the **Diagnostic** tab, record the Max interferogram value in the logbook and then select **Align.**

   3.) A window appears advising that Bench Alignment is in progress and should take 2-3 minutes to complete. Record the Max interferogram value after alignment in the logbook and select **OK**.

Instrument Shutdown:

1. Exit OMNIC by selecting the red X in the upper right corner of the window.

2. Close the laptop.

Collecting Background and Sample Spectra:

Before collecting a spectrum ensure that the OMNIC window is active, the correct Experiment Method is selected ("**iTR Diamond**" for ATR experiments) and that the Bench Status indicator is a green check mark. See **Startup** Section for reference.

1. If a previous scan is displayed in the OMNIC window, clear it before starting a new scan by clicking the small gray **X** just above the Bench Status indicator.

2. To collect sample scans, select the **"Col Smp"** icon. As a background is required before every sample scan, a prompt to collect a background will appear. Select **OK** when ready.

3. When the background is complete a prompt to collect the sample spectrum will appear. Select **OK** when ready.

4. When the sample scan is completed a prompt for spectrum title will appear. Overwrite the date and time with sample identifier (case and item number for example). Select **OK**.

5. Select **YES** to add the spectrum to a new window.

6. Select **OK** if prompted for a window title.

7. To save a file, select **FILE** and **SAVE AS** from the toolbar.

8. Overwrite the default file name with the next data file name from the logbook and select **SAVE**. Record the appropriate information in the logbook.

Data Analysis:

To perform a library search

1. Select **"Lib Setup"** icon from the toolbar. The parameters should read as follows:

Search Libraries tab	*In-House v2 (or whichever libraries are desired)*
Search Results tab	*Configure search results button selected* *Search type: Correlation* *List compounds with match values above: 0 (default)* *Maximum number of compounds in list: 10 (default)* *Number of library spectra to display: 1 (default)* *Show match values selected*
Search Regions tab	*Use full spectral range selected*

NOTE: This is not necessary with every run since the parameters will not change from run to run.

2. Select **"Search"** to perform the search.

To perform a spectrum subtraction

As FTIR spectrometry produces a combined spectrum for all components in a sample, the analyst may need to remove the contribution from a major component to identify another component of interest. This is usually indicated from the search results where the first match accounts for some but not all of the sample peaks (ex. dimethylsulfone with methamphetamine). The analyst can use the Subtract function to remove these indicated substances and then perform a new search on the resultant spectrum as follows:

1. After running the sample, perform a Search to determine what should be subtracted. (Usually the first match will be a good place to start.) Close out of the search window and return to the original sample spectrum.

2. Select "**Lib Mgr**" from the toolbar.

3. Open "**Search Libraries**" and select the library (In-House v2, HR Georgia State Forensic Drug, etc.) which contains the spectrum to be subtracted.

4. Click on the "**Search for Text**" tab.

5. Type in the name of the substance to be subtracted.

6. Double click on the substance to be subtracted.

7. Add to the same window as the original sample spectrum.

8. Close Library Manager.

9. Select "**Stack Spe**" from the toolbar to separate the two spectra.

10. Click in the half of the window with the sample spectrum.

11. Hold down the Control key and click in the other half of the window with the spectrum to be subtracted. (There should be a message in the Information bar which says "Two spectra selected").

12. Select "**Process**" from the toolbar and click on "**Subtract**".

13. Three spectra should appear: The original sample spectrum, the spectrum to be subtracted, and the result of the subtraction (this should be on the bottom).

14. The subtraction factor can be adjusted by scrolling up or down on the Factor button on the left side of the screen. The Coarse and Finer buttons may be used to increase or decrease the factor.

15. Once satisfied with the results, click on "Add to new window" and perform a new Search.

To print a spectrum scan or library search

1. Select **Report** and **Template** from the menu toolbar.

2. The default should be **"HFSC TEMPLATE"**. If so, then select **Close**. If not, then highlight it and choose **Select**.

3. Select the "**Prev Rpt**" icon and prompts will appear to enter the **Analyst**, then **Description**, and then **Case #**.

4. The screen will display a preview of the report with the information entered. If the information is correct, select **Print** and **Close**. If the information is not correct, then select **Close** without printing and repeat steps 3 and 4.

Instrument Performance Check (performed quarterly or as needed):

Follow routine **Startup** procedures before performing an Instrument Performance Check.

1. Remove the ATR accessory. Install the standard sample holder and cover. A window will appear to confirm the accessory change and that the **"Transmission E.S.P."** Experimental Method has been loaded. Select **OK**. A Smart Accessory Test window will appear. A green check mark verifies the accessory change, select **OK** and continue.

2. Allow the system to equilibrate for at least 15 minutes.

3. Start ValPro performance check by selecting **Analyze** and **ValPro Qualification** from the menu toolbar. Go to the drop down menu at the top of the screen and select "**Nicolet iS50 system with DTGS in built-in position KBr-EP**".

4. Select **Options**. The default parameters should read as follows:

   Select "Print Report" and "Display Report" under the Reports tab
   Select "Enable Validation Wheel" under the Configure tab
   Select "Save Spectra" under the Results tab
   Select "Nicolet iS50 system with DTGS in built-in position KBr-EP" under the Qualification Tests tab

   Select **OK**.

5. Select **Qualify** and at the prompt to clear the sample compartment select **OK** when ready.

6. Upon completion, the "**OMNIC ValPro Qualification Report**" screen will appear. A report will automatically be sent to the printer. If it does not, select **Print** and then **Close.**

7. Press Close to return to the OMNIC software.

8. Record the instrument performance check results in the logbook and store the printouts in the appropriate location. The test results obtained by utilizing the ValPro performance checks are compared to prior results to verify that the system is working consistently over time. If any problems occur or the report obtained indicates failure of one or more tests based on the given factory pass-fail range, consult the assigned analyst(s) for potential causes and corrective recommendations. If these do not correct the problem, the instrument should be taken out of service until corrective action is taken and the problem corrected.

9. Remove the standard sample holder and cover. Install the ATR accessory and select **OK** to confirm the **"iTR Diamond"** Experimental Method. A Smart Accessory Test window will appear. A green check mark verifies the accessory change, select **OK**.

ATR Correction:

In the ATR technique, the depth of penetration (that is, the effective pathlength) of the infrared beam varies as a function of the wavelength of light: The longer wavelengths (lower frequencies) penetrate the sample more deeply than do the shorter wavelengths (higher frequencies). As a result, the bands at lower frequencies are much stronger than those at higher frequencies. This skewing of band intensities causes problems when searching a sample spectrum against a library of spectra collected using standard transmission / absorbance techniques, since the bands have different relative intensities and band positions.

The Experimental Method used for ATR sampling includes a correction for this effect which multiples the sample spectrum by a wavelength-dependent factor to adjust the relative band intensities. The resulting spectrum has bands more like those in a typical absorbance spectrum and can be visually compared with absorbance spectra or searched against a library of absorbance spectra.

Carbon dioxide correction:

In a typical FTIR experiment the sample spectrum is ratioed against a background spectrum that contains all of the spectral characteristics of the instrument. These characteristics include absorptions due to any atmospheric water vapor or carbon dioxide. Ratioing ensures that the sample spectrum contains information that is characteristic only of the sample.

Since sample and background spectra are collected separately, the water and carbon dioxide absorptions may not be exactly the same in both spectra. This can result in positive (or negative) peaks in the water (3,800 and 1,600 cm^{-1}) and carbon dioxide (2,350 and 668 cm^{-1}) regions of the ratioed sample spectrum. These residual peaks may cause problems when a spectrum is searched against a library.

To remove excess carbon dioxide contributions from a spectrum, use the following steps for an already saved data file:

1. Select **Region tool** (the second icon in the lower left of screen).

2. Using the cursor, point to where you want the region to start and press and hold down the mouse button.

3. While holding down the mouse button, move the pointer to where you want the region to end. Release mouse button.

4. Select **Process** and **Straight line** to fill the highlighted region with a solid line.

5. Click **Selection tool** (the first icon in the lower left of screen) to turn off the **Region tool**.

Search Type Algorithms:

Correlation
Normally gives the best results and is recommended for most applications. The algorithm removes any effect of offset in the unknown spectrum, thus eliminating the effects of baseline variation. This is the usual method used.

Absolute difference
Puts more weight on the small differences between the unknown spectrum and library spectra. This means that impurities will have a larger effect on the search results.

Squared difference
Emphasizes the large peaks in the unknown spectrum. This algorithm may be used when identifying a noisy spectrum.

Absolute derivative
Gives small peaks and peak shifts an increased effect on the search results. The algorithm removes any differences between the unknown and library spectra caused by an offset in the unknown spectrum. This algorithm is useful when you want to emphasize peak positions rather than peak intensities. This algorithm may be used when identifying a spectrum with a tilted baseline.

Squared derivative
Emphasizes large peaks as well as peak shape. The algorithm removes any differences between the unknown and library spectra caused by an offset in the unknown spectrum. This algorithm works well with spectra of poor quality.

MODIFICATION SUMMARY

ISSUE DATE	CHANGE
Current Version	New document for new instrumentation

End of Document